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Jul 12, 1988

DERWENT-ACC-NO: 1988-232659
DERWENT-WEEK: 198833
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TITLE: Oxidn. resistant permanent magnet - comprises coating contg. titanium and at least one of neodymium, iron boron and oxygen on neodymium-iron-bor on magnet

PATENT-ASSIGNEE:

ASSIGNEE	CODE
TAIYO YUDEN KK	TAIO

PRIORITY-DATA: 1986JP-0311432 (December 30, 1986)

PATENT-FAMILY:

PUB-NO	PUB-DATE	LANGUAGE	PAGES	MAIN-IPC
JP <u>63168009</u> A	July 12, 1988		008	

APPLICATION-DATA:

PUB-NO	APPL-DATE	APPL-NO	DESCRIPTOR
JP63168009A	December 30, 1986	1986JP-0311432	

INT-CL (IPC): B22F 3/24; C23C 18/02; C23C 30/00; H01F 7/02; H01F 41/02

ABSTRACTED-PUB-NO: JP63168009A

BASIC-ABSTRACT:

A coating of a cpdr. contg. Ti and at least one of Nd, Fe, B and O is provided on surface of Nd-Fe-B magnet.

USE/ADVANTAGE - As the produced permanent magnet has a chemically stable cpd. coat on the surface, it has high resistance to oxidn. Deterioration of magnetic characteristics with time is reduced.

CHOSEN-DRAWING: Dwg.0/1

TITLE-TER MS: OXIDATION RESISTANCE PERMANENT MAGNET COMPRISE COATING CONTAIN TITANIUM ONE NEODYMIUM IRON BORON OXYGEN NEODYMIUM IRON BORON MAGNET

DERWENT-CLASS: L03 M13 P53 V02

CPI-CODES: L03-B02A; M13-B;

EPI-CODES: V02-E01; V02-H04;

SECONDARY-ACC-NO:

CPI Secondary Accession Numbers: C1988-104260

Non-CPI Secondary Accession Numbers: N1988-176797

L Number	Hits	Search Text	DB	Time stamp
1	659	(148/101).CCLS.	USPAT; EPO; JPO; DERWENT; IBM_TDB	2002/08/08 11:51
2	218	(148/247).CCLS.	USPAT; EPO; JPO; DERWENT; IBM_TDB	2002/08/08 11:49
3	257	(148/284).CCLS.	USPAT; EPO; JPO; DERWENT; IBM_TDB	2002/08/08 11:49
4	240	(148/279).CCLS.	USPAT; EPO; JPO; DERWENT; IBM_TDB	2002/08/08 11:49
5	1359	((148/101).CCLS.) ((148/247).CCLS.) ((148/284).CCLS.) ((148/279).CCLS.)	USPAT; EPO; JPO; DERWENT; IBM_TDB	2002/08/08 11:51

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Search Results - Record(s) 1 through 1 of 1 returned.☐ 1. Document ID: JP 07230906 A

L1: Entry 1 of 1

File: DWPI

Aug 29, 1995

DERWENT-ACC-NO: 1995-334129

DERWENT-WEEK: 199543

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TITLE: Permanent magnet for OA appts and precision instrument - has modified silica compound coating provided on neodymium-iron@-boron@ system alloy surface

PRIORITY-DATA: 1994JP-0019653 (February 16, 1994)

PATENT-FAMILY:

PUB-NO

PUB-DATE

LANGUAGE

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JP 07230906 A

August 29, 1995

009

H01F001/053

INT-CL (IPC): H01 F 1/053; H01 F 41/02

ABSTRACTED-PUB-NO: JP07230906A

BASIC-ABSTRACT:

The permanent magnet (1) is formed by applying an organically modified silicon compound coating (3) on a Nd-Fe-B alloy magneto surface (2). The silicon compound has a precursive substance and also includes a methacrylate compound and an epoxy compound usually the precursive substance is a vinyl compound. The precursive substance is agitated with diluted acid. Then hydrolysis and polycondensation is carried out for sol formation. Then, hardening process is carried out by heat application and the organically modified compound applied onto the magnetic alloy surface.

ADVANTAGE - Excels in magnetic characteristics and magnetic corrosion resistance. Facilitates low cost mfg. Realises longer life span. Avoids troublesome process like painting and plating.

Full	Title	CIT.1	REV.1	CLS.1	REF.1	SEQ.1	ATT.1
RAW.1	IP.1						

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Term	Documents
"07230906".DWPI.	1
07230906S	0
"07230906".DWPI.	1
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CLAIMS

[Claim(s)]

[Claim 1] The permanent magnet characterized by forming an organic denaturation silica system coat in the front face of a Nd-Fe-B system alloy magnet.

[Claim 2] The permanent magnet according to claim 1 with which thickness of an organic denaturation silica system coat is characterized by being 0.1-500 micrometers.

[Claim 3] The permanent magnet according to claim 1 or 2 characterized by an organic denaturation silica system coat being a hardening coat formed from at least one sort of a silica system precursor and the organic system precursor chosen from the methacrylate system, the epoxy system, and the vinyl system.

[Claim 4] The permanent magnet according to claim 1, 2, or 3 with which an organic denaturation silica system coat is characterized by the bird clapper from 30-70 mol % and 30-70 mol [of organic system precursor components] % of silica system precursor components.

[Claim 5] The permanent magnet according to claim 1, 2, 3, or 4 with which an organic denaturation silica system coat is characterized by the bird clapper from a silica-methacrylate system hardening coat.

[Claim 6] Into at least one sort of a silica system precursor and the organic system precursor chosen from the methacrylate system, the epoxy system, and the vinyl system of mixture By adding and agitating acid water, and carrying out heat-curing processing, after being immersed and pulling up a Nd-Fe-B system alloy magnet in hydrolysis and the formed gel which carried out the polycondensation The manufacture method of the permanent magnet characterized by forming an organic denaturation silica system coat in the front face of the aforementioned Nd-Fe-B system alloy magnet.

[Claim 7] The manufacture method of the permanent magnet according to claim 6 characterized by adjusting the thickness of the organic denaturation silica system coat formed by controlling the concentration of gel, and/or the raising speed of a Nd-Fe-B system alloy magnet in the range of 0.1-500 micrometers.

[Claim 8] The manufacture method of the permanent magnet according to claim 6 or 7 characterized by forming gel using the 30-70 mol mixture (% and 30-70 mol % of organic system precursor components) of silica system precursor components.

[Claim 9] The permanent magnet according to claim 6, 7, or 8 characterized by forming gel from the mixture of a silica system precursor component and an organic methacrylate system precursor.

[Claim 10] The manufacture method of the permanent magnet according to claim 6, 7, 8, or 9 characterized by removing moisture from the aforementioned gel coat by infrared irradiation or vacuum heating in advance of heat-curing processing of the gel coat formed in the front face of a Nd-Fe-B system alloy magnet.

[Claim 11] gel -- the mixture of a silica system precursor component and an organic system precursor component -- in addition, at least one sort of the solid-state powder chosen from a zinc chromate, an iron oxide, a mica, a silica, titanium oxide, and the zirconia -- 2-40-mol % -- the manufacture method of the permanent magnet according to claim 6, 7, 8, 9, or 10 characterized by containing

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DETAILED DESCRIPTION

[Detailed Description of the Invention]

[0001]

[Industrial Application] This invention relates to a permanent magnet and its manufacture method. They are efficient and a thing about the method of manufacturing to a low cost in more detail about the permanent magnet excellent in magnetic properties and corrosion resistance, and this permanent magnet.

[0002]

[Description of the Prior Art] A rare earth iron system sintered-alloy magnet, especially a Nd(neodim)-Fe(iron)-B (boron) system alloy magnet are excellent in magnetic properties, and since it is a low cost, they are widely used in fields, such as OA equipment and a precision mechanical equipment.

[0003] However, since the Nd-Fe-B system alloy magnet made iron the principal component, it tended to generate rust in response to the influence of the moisture in air, or oxygen, was inferior in corrosion resistance, and had the fatal fault that an endurance life was short.

[0004] Then, in order to intercept in the former that moisture etc. invades from the front face of a Nd-Fe-B system alloy magnet and to improve the corrosion resistance of this alloy magnet Although the method of carrying out plating covering of the corrosion-resistant metal coats, such as the method of carrying out paint covering of the organic system resins, such as an epoxy system, and nickel, etc. was adopted as the front face of the aforementioned alloy magnet By these methods, adhesion with the aforementioned alloy magnet, the aforementioned paint coat, or a plating coat was bad, and since the mechanical strength was inferior, a magnet performance was not fully improvable.

[0005] In view of this actual condition, many corrosion resistance of a Nd-Fe-B system alloy magnet and methods of improving in accordance with a mechanical property are made in recent years. To the opening between the crystal grain of the ** aforementioned alloy magnet, for example, the linseed oil, a wax, a varnish, It is filled up with encapsulants, such as water glass, polyester resin, phenol resin, and an epoxy resin. How (JP,62-287004,A) to improve corrosion resistance by furthermore performing surface treatment, such as paint and coating, on the front face of this magnet, ** How (JP,63-168009,A) to improve corrosion resistance by heat-treating in an inert atmosphere after making organic titanium adhere to the front face of the aforementioned alloy magnet, And while preventing generation of big and rough crystal grain and attaining simplification of a process by forming the nature coat of silicon in the front face in the end of an alloy powder which is the material of the ** aforementioned alloy magnet with water glass etc., and subsequently fabricating a magnet using this end of an alloy powder The method (JP,3-295204,A) of enabling use in a high atmosphere of oxygen tension etc. is proposed.

[0006] However, by the above-mentioned ** and the ** method, the Nd-Fe-B system alloy magnet (permanent magnet) obtained by these methods was still inadequate about corrosion resistance, although just the mechanical property was improved, and large-sized equipment was needed, and moreover especially, in order to cause complication of a process, the problem that magnetic cost reduction could not be planned was included.

[0007]

[Problem(s) to be Solved by the Invention] This invention is attained as a result of inquiring in order to solve the trouble which the conventional permanent magnet mentioned above has.

[0008] Therefore, the purpose of this invention is about the permanent magnet excellent in magnetic properties and corrosion resistance, and this permanent magnet to offer [efficient and] the method of manufacturing to a low cost.

[0009]

[Means for Solving the Problem] In order to attain the above-mentioned purpose, the permanent magnet of this invention is characterized by forming an organic denaturation silica system coat in the front face of a Nd-Fe-B system alloy magnet.

[0010] The manufacture method of the permanent magnet this invention Moreover, a silica system precursor and a methacrylate system, Into at least one sort of the organic system precursor chosen from the epoxy system and the vinyl system of mixture It is characterized by forming an organic denaturation silica system coat in the front face of the aforementioned Nd-Fe-B system alloy magnet by adding and agitating acid water, and carrying out heat-curing processing, after being immersed and pulling up a Nd-Fe-B system alloy magnet in hydrolysis and the formed gel which carried out the polycondensation.

[0011]

[Function] The permanent magnet of this invention can demonstrate the corrosion resistance which fully prevented the invasion of moisture or oxygen on the front face of a Nd-Fe-B system alloy magnet by the organic denaturation silica system coat, and was excellent in it, holding the magnetic properties which were excellent in the Nd-Fe-B system alloy magnet since the organic denaturation silica system coat was formed, and can realize magnetic reinforcement.

[0012] Moreover, according to the manufacture method of the permanent magnet this invention, troublesome processes, such as the conventional paint and plating, are not needed, but the permanent magnet excellent in magnetic properties and corrosion resistance can be manufactured to efficient and a low cost according to the simple process of carrying out dipping post heating hardening processing of the magnet into gel.

[0013]

[Example] Hereafter, the permanent magnet of this invention and the example of the manufacture method are explained concretely, referring to a drawing.

[0014] Cross-section explanatory drawing in which drawing 1 shows an example of the permanent magnet of this invention, and drawing 2 are the process schematic diagrams showing the manufacture method of the permanent magnet this invention.

[0015] example **** shown in drawing 1 -- the permanent magnet 1 of this invention consists of a Nd-Fe-B system alloy magnet 2 and an organic denaturation silica system coat 3 formed in the front face

[0016] In the Nd-Fe-B system alloy magnet 2 as used in the field of this invention, the magnet which contains neodymium, iron, and boron at least is meant, and little content of the metallic element chosen from the rare earth elements chosen from a lanthanum, a cerium, plastic SEOJIUMU, a dysprosium, a holmium, and tele BIUMU besides these components, aluminum, titanium, vanadium, chromium, manganese, cobalt, nickel, a zirconium, niobium, molybdenum, the tantalum, and the tungsten can be carried out according to a request.

[0017] It is suitable for the rate of each component in this Nd-Fe-B system alloy magnet that there have 5-15-mol % and iron, and neodymium usually has boron in the 2-10-mol range of % 70-90-mol%.

[0018] In addition, the Nd-Fe-B system alloy magnet 2 prepares the raw material which can usually manufacture by the well-known method, for example, consists of the neodymium, the iron, the boron, and other metals of a predetermined rate, casts this mixture in meanses, such as high-frequency heating, under a high vacuum, it cracks, and carries out coarse grinding of the obtained ingot, and makes it with the end of an alloy powder. Next, a desired Nd-Fe-B system alloy magnet can be obtained by pulverizing the aforementioned end of an alloy powder using a jet mill etc., and sintering the Plastic solid which fabricated this impalpable powder all over the magnetic field under a high vacuum and an elevated temperature within a firing furnace.

[0019] Moreover, the organic denaturation silica system coat 3 is been the hardening coat of a silica system precursor and the organic system precursor chosen from the methacrylate system, the epoxy system, and the vinyl system especially formed from the methacrylate system precursor preferably at least one sort, and especially the thickness has 0.1-500 micrometers in the range of 0.1-50 micrometers.

[0020] And as for the organic denaturation silica system coat 3, it is desirable that it is especially the hardening coat which consists of 30-70 mol % of the above-mentioned silica system precursor components and 30-70 mol % of the above-mentioned organic system precursor components.

[0021] Here, a tetraethyl orthochromatic silicate etc. is mentioned as an example of the above-mentioned silica system precursor component.

[0022] Moreover, as an example of the methacrylate system precursor in the above-mentioned organic system precursor component, 3-glycyl oxy-propyltrimethoxysilane (GPMS) etc. is mentioned as an example of an epoxy system precursor, and ORGANO modification door RUKOKISHIDO, 3-metacryloxy chill trimethoxysilane (MPMS), etc. are mentioned for vinyl-triethoxysilane (VES) etc. as an example of a vinyl system precursor, respectively.

[0023] Thus, an organic denaturation silica system coat is formed in the front face of a Nd-Fe-B system alloy magnet, and the permanent magnet of invention of a bird clapper can demonstrate the corrosion resistance which fully prevented the invasion of moisture or oxygen by the organic denaturation silica system coat, and was excellent, with the magnetic properties held which were excellent in the Nd-Fe-B system alloy magnet, can realize magnetic reinforcement, and is very useful for uses, such as OA equipment and a precision mechanical equipment.

[0024] Next, according to drawing 2, one example of the manufacture method of the permanent magnet of this invention is explained in full detail.

[0025] In the manufacture method of this invention, a possible sol-gel method is easily adopted for the deposition of a coat as the formation method of the above-mentioned organic denaturation silica system coat 3.

[0026] That is, the mixture which mixed the silica system precursor and the organic system precursor at a desired rate can be used for the sol which consists of a silica system precursor and an organic system precursor first as ORUMOSHIRU of sol formation, it can add acid water, such as a hydrochloric acid, a nitric acid, and a sulfuric acid, to this, can fully agitate it, and can be easily formed by making hydrolysis and a polycondensation occur.

[0027] Here, on the occasion of formation of gel, it will be desirable % and to 30-70-mol consider [% and 30-70 mol] as 30-50-mol %/50 - 70-mol % preferably, and, less than [30 mol %], a sol will divide [a silica system precursor component] the rate of a silica system precursor component / organic system precursor component into two phases. Moreover, at more than 50 mol %, in order that a sol may gel for a short period of time, it is not-desirable.

[0028] Next, thickness can form a uniform organic denaturation silica system coat by putting the gel obtained above into arbitrary DIP coating equipments, putting this into thermal treatment equipments, such as oven, and performing heat-curing

processing at the temperature of about 100-200 degrees C for 5 to 15 hours, after carrying out being request time immersed of the Nd-Fe-B system alloy magnet (dipping) and pulling up it in gel.

[0029] In addition, it is desirable by controlling appropriately the concentration of the above-mentioned gel, and/or the raising speed of a Nd-Fe-B system alloy magnet to adjust the thickness of the organic denaturation silica system coat formed in the range of 0.1-500 micrometers.

[0030] Moreover, when many moisture is contained in gel, it is desirable to remove moisture from the aforementioned gel coat by infrared irradiation or vacuum heating in advance of heat-curing processing of the gel coat formed in the front face of a Nd-Fe-B system alloy magnet.

[0031] furthermore, at least one sort of the solid-state powder chosen from a zinc chromate, an iron oxide, a mica, a silica, titanium oxide, and the zirconia to the gel which consists of a silica system precursor component and an organic system precursor component -- 2-40-mol % -- the mechanical property of an organic denaturation silica system coat can be made into what was further excellent by adding In addition, after adding solid-state powder to gel in this case, it is desirable by performing ultrasonication etc. to promote the distributed system to the gel of solid-state powder.

[0032] In this way, according to the manufacture method of the above-mentioned permanent magnet this invention, troublesome processes, such as the conventional paint and plating, are not needed, but the permanent magnet excellent in magnetic properties and corrosion resistance can be manufactured to efficient and a low cost according to the simple process of carrying out dipping post heating hardening processing of the magnet into gel.

[0033] The example of an examination is given to below and the composition and the effect of this invention are explained further in full detail.

[0034] As a preparation Nd-Fe-B system alloy magnet of a <example of examination> (1) Nd-Fe-B system alloy magnet, the thing of the shape of a 3mmx10mmx50mm legislation bodily shape which consists of neodium 5 - 15-mol %, iron 70 - 90-mol %, boron 2 - ten-mol % of a rate was prepared.

[0035] (2) Preliminary mixture of adjustment tetraethyl orthochromatic silicate 13.44ml of a sol and the ORGANO modification door RUKOKISHIDO 34.50ml was carried out for 5 minutes, 0.60ml of hydrochloric acids and 6.0ml of water were added to this 35%, and it fully mixed. In 1 minute after a mixed start, mixture became transparent and generated heat quickly. The transparent sol was obtained by furthermore continuing mixture for about 20 minutes.

[0036] (3) The above-mentioned sol was put into DIP coating DIP coat equipment, and the Nd-Fe-B system alloy magnet attached in the electrode holder was pulled up the speed for 9cm/, after being immersed in the sol.

[0037] (4) Although the organic denaturation silica system coat started hardening immediately after pulling up from the heat-curing processing sol, after performing vacuum heat-treatment at 120 degrees C first for 3 hours and evaporating moisture, when heat-curing processing was performed in 160-degree C heat oven for 10 hours, the uniform silica-methacrylate system hardening coat of 10 micrometers of thickness was able to be formed. Let this permanent magnet be Sample A.

[0038] In addition, the nickel coat of 10 micrometers of thickness was formed in the front face of the same Nd-Fe-B system alloy magnet as the above by the usual electroplating for comparison. Let this permanent magnet be Sample B.

[0039] Furthermore, the epoxy resin coat of 20 micrometers of thickness was formed in the front face of the same Nd-Fe-B system alloy magnet as the above by the usual coating method. Let this permanent magnet be Sample C.

[0040] (4) About three kinds of permanent magnets obtained by the corrosion resistance evaluation above, the corrosion resistance test by the following conditions was performed, and the evaluation result was shown in Table 1.

[0041] ** Carry out the 500 cycles of this at a spalling-test room temperature-(5min) ->40-degree-C(1hr keeping)-(5min) ->120-degree-C(1hr keeping)-(5min) -> room temperature, and judge a result on the following criteria.

[0042] O A surface crack, ** -less all corrosion x Surface crack nothing, however corrosion advance somewhat.

[0043] ** 80-degrees C of high-humidity-examinations, and 95% of humidity, hr-500 times and judge a result on the following criteria.

[0044] O Some surface cracks, crack nothing of ** -less all corrosion ** front faces, however corrosion are advance x. There is a crack covering the whole front face and a corrosion region is also generated.

[0045] ** hr 1000 times at 120 degrees C of high temperature examinations, and judge a result on the following criteria.

[0046] O A surface crack, 35 degrees C of all corrosion 5 % of the weight brine of ** -less ** salt spray tests, 48hr (based on ASTM B-117)

It carries out and a result is judged on the following criteria.

[0047] O A surface crack, ** -less all corrosion x There is a crack covering the whole front face and a corrosion region is also generated.

[0048]

[Table 1]

サンプルNo.	A	B	C
熱衝撃試験	○	×	○
高湿度試験	○	△	×
高温試験	○	○	○
塩水噴霧試験	○	×	×
総合評価	○	×	×

The permanent magnet A of this invention is extremely excellent in corrosion resistance as compared with the conventional permanent magnets B or C so that clearly from the result of Table 1.

[0049]

[Effect of the Invention] As explained above, the permanent magnet of this invention can demonstrate the corrosion resistance which fully prevented the invasion of moisture or oxygen by the organic denaturation silica system coat, and was excellent, holding the magnetic properties which were excellent in the Nd-Fe-B system alloy magnet, since the organic denaturation silica system coat was formed in the front face of a Nd-Fe-B system alloy magnet, and can realize magnetic reinforcement.

[0050] Moreover, according to the manufacture method of the permanent magnet this invention, troublesome processes, such as the conventional paint and plating, are not needed, but the permanent magnet excellent in magnetic properties and corrosion resistance can be manufactured to efficient and a low cost according to the simple process of carrying out dipping post heating hardening processing of the magnet into gel.

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